

BRAMMER STANDARD COMPANY, INC.

Certificate of Analysis

BS Slag 3

Reference Material for Iron-making Slag

	Certified Value ¹	Estimate of Uncertainty ²	Certified Values ³	Certified Value ¹	Estimate of Uncertainty ²
Al	6.8	0.2	Al₂O₃	12.9	0.5
Ca	26.7	0.4	CaO	37.4	0.3
Fe	0.251	0.008			
K	0.67	0.02	K₂O	0.81	0.03
Mg	5.0	0.2	MgO	8.3	0.3
Mn	1.32	0.04	MnO	1.70	0.05
Na	0.19	0.02	Na₂O	0.26	0.02
S	0.82	0.03			
Si	17.4	0.5	SiO₂	37.3	0.3
Ti	0.37	0.01	TiO₂	0.62	0.02

Informational Values^{3,4}

C (0.038)

¹ For each element, the certified value listed is the present best estimate of the true value based on the mean of the weighted results of an interlaboratory testing program. See page 3 for more information on its calculation.

² For each element, the uncertainty listed is based on a statistical evaluation of the contributions of homogeneity and the interlaboratory testing program. See page 3 for more information on its calculation.

³ Values are given in weight percent. Values in brackets are reported by difference.

⁴ Values in parentheses are not certified and are provided for information only.

The requirements of ISO Guides 30, 31, and 35 were followed for the preparation of this Reference Material and certificate of analysis.

BS Slag 3 * Code for method Certified values listed as weight percent

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Analysis	*	Al	*	Al ₂ O ₃	*	Ca	*	CaO	*	Fe	*	K	*	K ₂ O	*	Mg
1	4	6.5333	4	12.3444	4	26.46	4	37.02	4	0.20	4	0.61	4	0.74	4	4.82
2	4	6.68	4	12.63	4	26.55822	4	37.16	4	0.215	4	0.66	4	0.80	4	4.96
3	4	6.779715	4	12.81	4	26.64	4	37.27	4	0.249	4	0.666	4	0.802	4	4.99
4	4	6.84	4	12.93	4	26.79238	4	37.48763	4	0.264467	4	0.67	4	0.81	4	5.0
5	4	6.93	4	13.09	4	26.87	4	37.59	4	0.28	4	0.675693	4	0.813933	4	5.017882
6	4	6.991415	4	13.21	4	26.92	4	37.67	4	0.291	4	0.697	4	0.840	4	5.09
7	4	7.02	4	13.26					4	0.291	4	0.73884	4	0.89	4	5.268073
8									4	0.30						
Average		6.82		12.88		26.71		37.37		0.2507		0.671		0.808		5.02
Std dev		0.26		0.49		0.36		0.25		0.0090		0.025		0.031		0.19
H		0.042091		0.070452		0.130861		0.175812		0.004195		0.007816		0.008845		0.033109
U ₁		0.26		0.49		0.38		0.31		0.0099		0.027		0.032		0.19
t-statistic		2.45		2.45		2.57		2.57		2.36		2.45		2.45		2.45
U ₂		0.64		1.20		0.98		0.79		0.023		0.065		0.078		0.47
U ₃		0.24		0.46		0.40		0.32		0.0083		0.025		0.029		0.18
Certified		6.8		12.9		26.7		37.4		0.251		0.67		0.81		5.0
Uncertainty		0.2		0.5		0.4		0.3		0.008		0.02		0.03		0.2
Tolerance		0.6		1.2		1.0		0.8		0.023		0.07		0.08		0.5

Analysis	*	MgO	*	Mn	*	MnO	*	Na	*	Na ₂ O	*	S	*	Si	*	SiO ₂
1	4	8.00	4	1.26	4	1.63	4	0.180	4	0.243	1	0.773	4	17.24765	4	36.89807
2	4	8.23	4	1.286716	4	1.661433	4	0.19	4	0.26	1	0.80	4	17.25	4	36.90
3	4	8.28	4	1.327	4	1.713	4	0.194291	4	0.2619	1	0.810	4	17.4	4	37.30
4	4	8.29	4	1.327	4	1.713449	4	0.21	4	0.28	1	0.81	4	17.48	4	37.40
5	4	8.32	4	1.329	4	1.716					1	0.823	4	17.5	4	37.43
6	4	8.44	4	1.33	4	1.717323					1	0.863333	4	17.54	4	37.53
7	4	8.734833	4	1.34	4	1.73					1	0.87	4	17.56174	4	37.57
8			4	1.343	4	1.734										
Average		8.32		1.317		1.700		0.1925		0.259		0.819		17.43		37.29
Std dev		0.31		0.047		0.060		0.0096		0.013		0.031		0.49		0.23
H		0.049325		0.012358		0.014797		0.003583		0.004278		0.008925		0.090748		0.175478
U ₁		0.32		0.048		0.062		0.010		0.014		0.032		0.50		0.29
t-statistic		2.45		2.36		2.36		3.18		3.18		2.45		2.45		2.45
U ₂		0.78		0.11		0.15		0.033		0.044		0.079		1.23		0.71
U ₃		0.29		0.040		0.052		0.016		0.022		0.030		0.46		0.27
Certified		8.3		1.32		1.70		0.19		0.26		0.82		17.4		37.3
Uncertainty		0.3		0.04		0.05		0.02		0.02		0.03		0.5		0.3
Tolerance		0.8		0.11		0.15		0.03		0.04		0.08		1.2		0.7

Analysis	*	Ti	*	TiO ₂
1	4	0.361	4	0.60217
2	4	0.361	4	0.602
3	4	0.365694	4	0.61
4	4	0.369	4	0.616
5	4	0.37	4	0.62
6	4	0.375	4	0.625
7	4	0.42	4	0.70
Average		0.373		0.622
Std dev		0.014		0.024
H		0.005357		0.007436
U ₁		0.015		0.025
t-statistic		2.45		2.45
U ₂		0.037		0.060
U ₃		0.014		0.023
Certified		0.37		0.62
Uncertainty		0.01		0.02
Tolerance		0.04		0.06

BS Slag 3 * Code for method Informational values listed as weight percent

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Analysis	*	C
1	1	0.02
2	1	0.037567
3	1	0.040
4	1	0.04
5	1	0.05
Average		0.04
Std dev		0.37
H		0.001523
U ₁		0.37
t-statistic		2.78
U ₂		1.030
U ₃		0.46
(Informational) (0.038)		

For each element, in accordance with the requirements of ISO Guides 34 and 35, an effort must be made to account for the effects on the certified value of the uncertainty estimate from homogeneity testing (H) and the uncertainties of the contributing laboratories. The average (A) is calculated using a weighted mean where the reciprocal of the square of each laboratory's combined uncertainty (C_L), calculated from its standard deviation (S_L) and its uncertainty estimate (U_L), is used as the weight (W_L) for its mean (M_L). The standard deviation (S) is calculated as the square root of the reciprocal of the sum of the weights. U₁ is the combined uncertainty from homogeneity and labs. U₂ is U₁ multiplied by the coverage factor (95 % t-statistic). U₃ is U₂ divided by the square root of the number of determinations (n). Thus:

$$C_L = \sqrt{S_L^2 + U_L^2} \quad W_L = \frac{1}{C_L^2} \quad A = \frac{\sum_{i=1}^n W_L M_L}{\sum_{i=1}^n W_L} \quad S = \frac{1}{\sqrt{\sum_{i=1}^n W_L}} \quad U_1 = \sqrt{H^2 + S^2} \quad U_2 = t \times U_1 \quad U_3 = \frac{U_2}{\sqrt{n}}$$

All but the final reported values are taken to two significant figures as determined by each quantity's uncertainty estimate. The final reported Uncertainty is U₃ rounded to one significant figure and represents the half width of the 95 % confidence interval for the **Certified** value. The final reported **Certified** value is A rounded to the same decimal place as the Uncertainty. The Uncertainty is a measure of the quality of the **Certified** value.

The Tolerance is a measure of the expected performance of an analysis. This involves further expanding the sample uncertainty to include instrument and operator uncertainty, for those without access to such calculations.

For further information regarding the confidence interval for the certified value see ISO Guide 35:2006 section 6.

Analytical Method Codes:

- | | | |
|---------------------------|---------------------------|---------------------------|
| 1 Combustion (ASTM E1019) | 7 Photometric | 13 Titrimetric |
| 2 Fusion (ASTM E1019) | 8 Flame Atomic Absorption | 14 DCP Atomic Emission |
| 3 Spark Atomic Emission | 9 GF Atomic Absorption | 15 HG Atomic Fluorescence |
| 4 ICP Atomic Emission | 10 X-Ray Fluorescence | 16 Difference |
| 5 ICP Mass Spectrometry | 11 GD Atomic Emission | |
| 6 Gravimetric | 12 GD Mass Spectrometry | |

ICP = Inductively Coupled Plasma GF = Graphite Furnace GD = Glow Discharge
DCP = Direct Current Plasma HG = Hydride Generation

Laboratory

Laboratory Testing, Inc.

Location

Hatfield, PA

Registrar

PRS/Nadcap

Accreditation

17025

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Brammer Standard Company, Inc.
Analytika, Ltd.
Andrew S. McCreath & Son, Inc.
Hoesch Stahl
Inland Steel
US Steel Research Laboratory
LECO Corporation

Houston, TX
Prague, Czechoslovakia
Harrishurg, PA
Dortmund, Germany
Chicago, IL
Monroeville, PA
St. Joseph, MI

Nadcap = National Aerospace and Defense Contractors Accreditation Program
PRI = Performance Review Institute

Analysis: Chemical analyses were made on samples taken from bulk powder material. The original participating laboratories normally followed the requirements of ISO Guide 25. The laboratories participating in the stability testing followed the requirements of ISO Standard 17025. Methods of analysis used were those listed on page 2.

Homogeneity: This Reference Material (RM) was tested for homogeneity using ASTM Standard Method E826 and found acceptable.

Validity statement: ISO Guide 31 states that the certification should contain an expiration date for all materials where instability has been demonstrated or is considered possible, after which the certified value is no longer guaranteed by the certifying body. The certification of BS Slag 3 is valid for 20 years from the date of this certificate. The certification is nullified if this RM is damaged, contaminated, or otherwise modified.

Storage: This RM will remain stable for twenty years, provided that the bottle remains sealed and is stored in a cool, dry atmosphere. When the bottle has been opened, the lid should be secured immediately after use. If the contents should become discolored (eg. Oxidized) due to atmosphere contamination, they should be discarded.

Source: This material was produced by USX Engineer & Consultants, Inc., Monroeville, PA. The material was made in a blast furnace.

Form: This RM is available only in the form of minus 200 mesh powder in 100 gram containers.

Use: This RM is intended for use in x-ray spectrometric and solution methods of analysis. Refer to ISO Guide 33 for information about the use of Certified Reference Materials.

Sample Preparation: For best analytical results, use the same method for preparing the analytical surface on all reference materials as used for production specimens.

Certificate Number: The unique identification number for this certificate of analysis is REVSslag 3-031517. You may obtain information on revisions of certificates from the internet at www.brammerstandard.com.

Safety Notice: A Safety Data Sheet (SDS) is not required for this material. This material will not release or otherwise result in exposure to a hazardous chemical, under normal conditions of use. Inquiries concerning this Reference Material should be directed to:

Brammer Standard Co., Inc. **Phone: (281) 440-9396** **Web: www.brammerstandard.com**
14603 Benfer Road
Houston, Texas 77069-2895 USA **Fax: (281) 440-4432** **Email: contact@brammerstandard.com**

Brammer Standard Company, Inc., is accredited by the American Association For Laboratory Accreditation (A2LA) to ISO Guide 34 as a Reference Material Producer for the production of Certified Reference Materials and Reference Materials (Certificate Number 656.02)

Brammer Standard Company's Chemical Laboratory is accredited by A2LA to ISO Standard 17025. (Certificate Number 656.01)

By Certificate Number 10539, the Quality System of Brammer Standard Company, Inc., is registered to ISO 9001:2008 by National Quality Assurance (NQA), U.S.A.

The scopes of accreditation are listed on the website: www.brammerstandard.com

Expiration: 20 years from date of certification, which is March 15, 2037. This material is valid until that date.

Revision: This reference material was originally certified as a reference material on September 24, 1992. The documents validity for Brammer Standard Company powder products is 20 years. Additional interlaboratory testing was performed in 2016 to prove stability and has been included on this certificate. C has been changed from certified to informational. Revised values for all elements except Al₂O₃, Ca, K, K₂O, Mg, MgO, Na, and Na₂O are presented.

References:

Versions used were those available at the time of testing and characterization

- E826 Standard Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry
- E1019 Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques
- E1806 Standard Practice for Sampling Steel and Iron for Determination of Chemical Composition

- ISO Standard 17025:2005 General requirements for the competence of testing and calibration laboratories
- ISO Standard 9001:2008 Quality Management Systems - Requirements
- ISO Guide 30:2015 Terms and definitions used in connection with reference materials + 2008 amendment
- ISO Guide 31:2015 Reference materials - Contents of certificates and labels
- ISO Guide 33:2015 Uses of certified reference materials
- ISO Guide 34:2009 General requirements for the competence of reference material producers
- ISO Guide 35:2006 Reference Materials - General and statistical principles for certification

ASTM documents available from ASTM, 100 Barr Harbor Dr., West Conshohocken, Pa 19428.

ISO Guides and Standards available from Global Engineering - www.global.ihs.com

Other useful documents available from NIST, U.S. Department of Commerce, Gaithersburg, MD 20899.

NIST Special Publication 260-100, Handbook for SRM Users

NIST Special Publication 829, Use of NIST Standard Reference Materials for Decisions on Performance of Analytical Chemical Methods and Laboratories

Certified by: _____ on March 15, 2017.

Beau R. Brammer
President